

QUARTZ CRYSTAL MICROBALANCE SYSTEMS FOR SHUTTLE CONTAMINATION MEASUREMENTS *

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ABSTRACT

The adsorption of contamination on a surface is highly temperature dependent. In order to conduct fundamental studies of contamination on Shuttle, surface temperatures must be controlled over a range common to the space environment. Two different types of temperature controlled quartz crystal microbalance systems using active and passive cooling have been developed to measure contamination as a function of temperature. The active system uses thermoelectric devices for automatic temperature control. Its purpose is to measure volatile and low-volatile contaminants, such as, lubricants and RTV's. The system consists of a controller and five remotely located 15-MHz optically-polished quartz crystal mass sensors. Each sensor can be set by ground command at five predetermined temperatures between -60 and $+80^{\circ}\text{C} \pm 1^{\circ}\text{C}$ or free run. The frequency change of each sensor is readout digitally to 1 Hz giving a contamination loading sensitivity of $1.56 \times 10^{-9} \text{ g/cm}^2$. The passive system is radiatively cooled and operates at cryogenic temperatures. Its purpose is to measure high-volatile contaminants, such as, water vapor. It consists of a controller and sensor head. The sensor head contains two removable sensors. One sensor operates below the freezing point of water and monitors contamination including water vapor. The other sensor is heated above the freezing point of water and monitors the contamination background. The temperature of each sensor is monitored between -153 and $+37^{\circ}\text{C} \pm 1^{\circ}\text{C}$. The mass sensitivity and frequency readout are the same as for the active system.

1.0 INTRODUCTION

When the Shuttle approaches orbital altitude, the atmospheric density will drop to low levels and the mean-free-path for gases will become larger than the dimension of the craft. At this point, a cloud of Volatile Condensable Material (VCM) will begin to envelope the Shuttle. This

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cloud consists primarily of the outgassing of materials used on its construction, desorption of surface contamination, rocket exhaust and waste dumps. Most of the particles in this induced contamination cloud will stream off radially into space with no adverse affect on surfaces. A fraction of these particles, though, will strike and contaminate various sensitive surfaces.

Surfaces can be contaminated by direct line of sight to the source, by redirection of contamination particles, by particle-particle interactions with the upper atmosphere and by particles of contamination that leave the Shuttle and vaporize on exposure to the sun. Today, I would like to describe two types of quartz crystal microbalance systems that we have developed for the NASA Marshall Space Flight Center to measure surface contamination on Shuttle. One system is a Temperature-Controlled Quartz Crystal Microbalance (TQCM) and the other is a Cryogenic Quartz Crystal Microbalance (CQCM).

2.0 SURFACE CONTAMINATION

The contamination of surfaces in space is affected by many parameters. Some of these parameters are temperature, type of material, condition of surface, exposure to solar radiation, sputtering¹ by the upper atmosphere, angle of incidence and intensity of the contamination flux. At altitudes beyond the magnetosphere, charging of the surface and ionization of the contaminate flux by the solar plasma can lead to the enhancement of contamination by mutual attraction.

Of all the various parameters affecting contamination, the temperature of the surface is dominant. This can be seen by going to extremes. If the surface temperature is steadily increased, the contaminants sticking coefficients will decrease and the desorption rates will increase resulting in low-levels of contamination. If the temperature is steadily decreased to values below the freezing points of the contaminants, the sticking coefficient will increase and desorption will decrease resulting in high-levels of contamination.

For purposes of a QCM contamination experiment, it is useful to divide contaminants into three broad groups depending upon the contaminants desorption activation energy, E , (cal/g-mol).

The first group is the high-volatiles for which $E < 10,000$ and covers materials, such as, H_2O and CO_2 . The temperature of a QCM must be steadily decreased below about $-130^\circ C$ to temperatures approaching liquid helium before these VCM's freeze out and make accurate measurements possible.

The second group are the volatiles for which $10,000 < E < 25,000$.

This range covers, for example, various types of lubricants and conformal coatings. Here, cooling of a QCM should start at about +30°C and be steadily decreased before measurements can be made.

The third group are the low-volatiles for which $E > 25,000$. This group covers, for example, epoxies, RTV's and rocket exhaust materials. A QCM operating at the ambient spacecraft temperature will usually give good results for these VCM's.

3.0 TQCM SYSTEM

3.1 TQCM Design

The TQCM System was developed to measure the flux of volatile and low-volatile contamination on Shuttle and is similar in design to the TQCM constructed for laboratory measurements².

The TQCM System is shown in Fig. 1. It consists of a controller and five electronics, sensors and heat sinks.

The mass sensors are instrumented with high-sensitive 15-MHz crystals with a mass sensitivity of 1.56×10^{-9} g/cm²-Hz. These crystals are over two times more sensitive than 10-MHz crystals presently being used in Faraday Laboratories QCM's³. The frequency of each sensor is readout digitally to ± 1 Hz. The crystals are optically polished and coated with MgF₂ to simulate an optical surface. Each sensor can be set by ground command at five predetermined temperatures between -60 and +80°C $\pm 1^\circ$ C or free run to obtain minimum operating temperature. The minimum temperature is 80°C below ambient. Maximum sensor loading is approximately 3×10^{-4} g/cm². If maximum loading is reached, the sensors can be cleaned by commanding the TQCM to +80°C. The operating temperatures of each sensor are monitored between -103 and +87°C.

By pointing the sensors in different directions, the following contamination studies can be made.

- 1) Temperature dependence
- 2) Adsorption and desorption rates
- 3) Spatial distribution
- 4) Time decay constants
- 5) Diurnal variations
- 6) Effects of solar irradiation
- 7) Upper atmosphere interactions
- 8) Sputtering

The spatial distribution with time and temperature will be defined by the output of the five separate TQCM sensors as the sensors are periodically

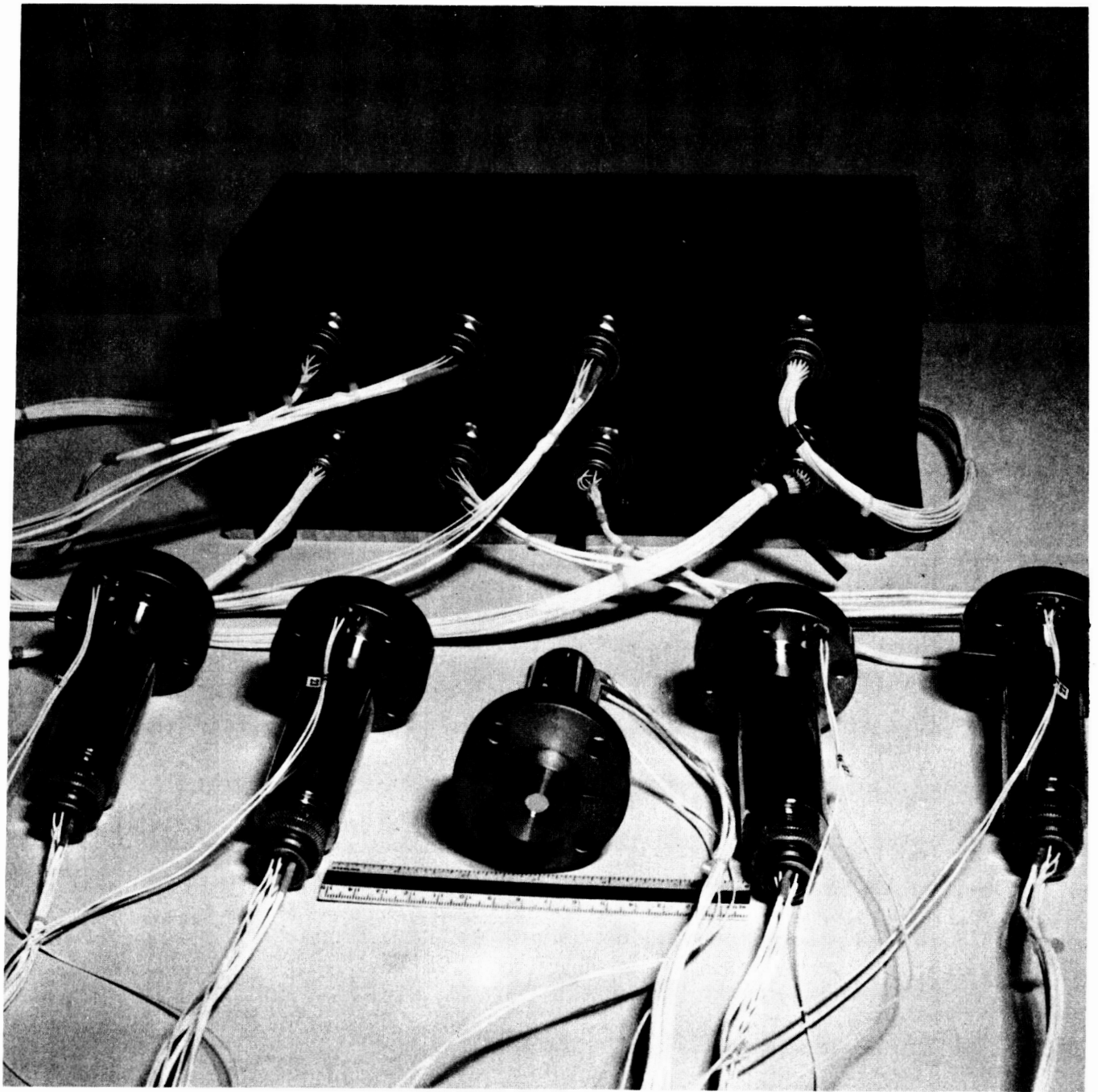


Fig. 1 TQCM System

commanded to various temperatures. At temperatures above +30°C, the low-volatile contaminants will be measured. Below +30°C, volatile materials will be measured. Computer programs will be written for "quick look" data reduction and for data analysis of the measurements to separate out the various contaminant species.

The mechanical specifications of the TQCM Controller are shown in Fig. 2. This drawing is taken from a large design print and the Controller dimensions are in inches (19.05 x 16.51 x 33.02 cm) to facilitate its machining.

The TQCM Electronics (3.18 by 11.75 cm), Heat Sink (6.35 by 1.9 cm) and Sensor (2.92 by 2.79 cm) are shown in Fig. 3.

The heat sink is gold plated aluminum for lightness. Its temperature is monitored by a Pt thermometer between -83 and +107°C \pm 1°C.

The electronics makes use of new low-power integrated circuits⁴. Its power dissipation is only 15 mW as compared to 250 mW in earlier units and significantly reduces the power load to the heat sink. The electronics provides circuits to drive the mass sensor and a high-gain low-impedance output amplifier for remote operation.

The removable mass sensor is designed for ease of replacement by loosening the lock and set screws on the heat sink. The crystal set is also replaceable. The precision Pt thermometer provides temperature readout to 0.5%. A specially designed two-stage thermoelectric device cools the crystals. It operates at \pm 7 Vdc for heating or cooling as compared to \pm 1.5 Vdc for laboratory devices for improved power supply efficiency.

The TQCM Controller Block Diagram is shown in Fig. 4. Separate high and low-power DC/DC supplies provide power for the thermoelectric devices and temperature control and sensor electronics. The frequency counter reads out TQCM frequencies to 65,535 Hz to \pm 1 Hz. By observing each time the counter resets itself, the full 300 kHz loading range of the crystal sensor can be attained.

The five remotely located sensor electronics are connected at J3-J7 and heat sinks at J8.

3.2 TQCM Operation

The TQCM commanded modes of operation are given in Table 1. The TQCM operates at +28 \pm 4 Vdc. Minimum power is 1.85 W with all QCM's ON and sensors operating at ambient temperatures. Maximum power is 71.12 W with all QCM's and all thermoelectric devices ON for cooling of the sensors.

The TQCM Controller is designed to maintain any sensor opera-

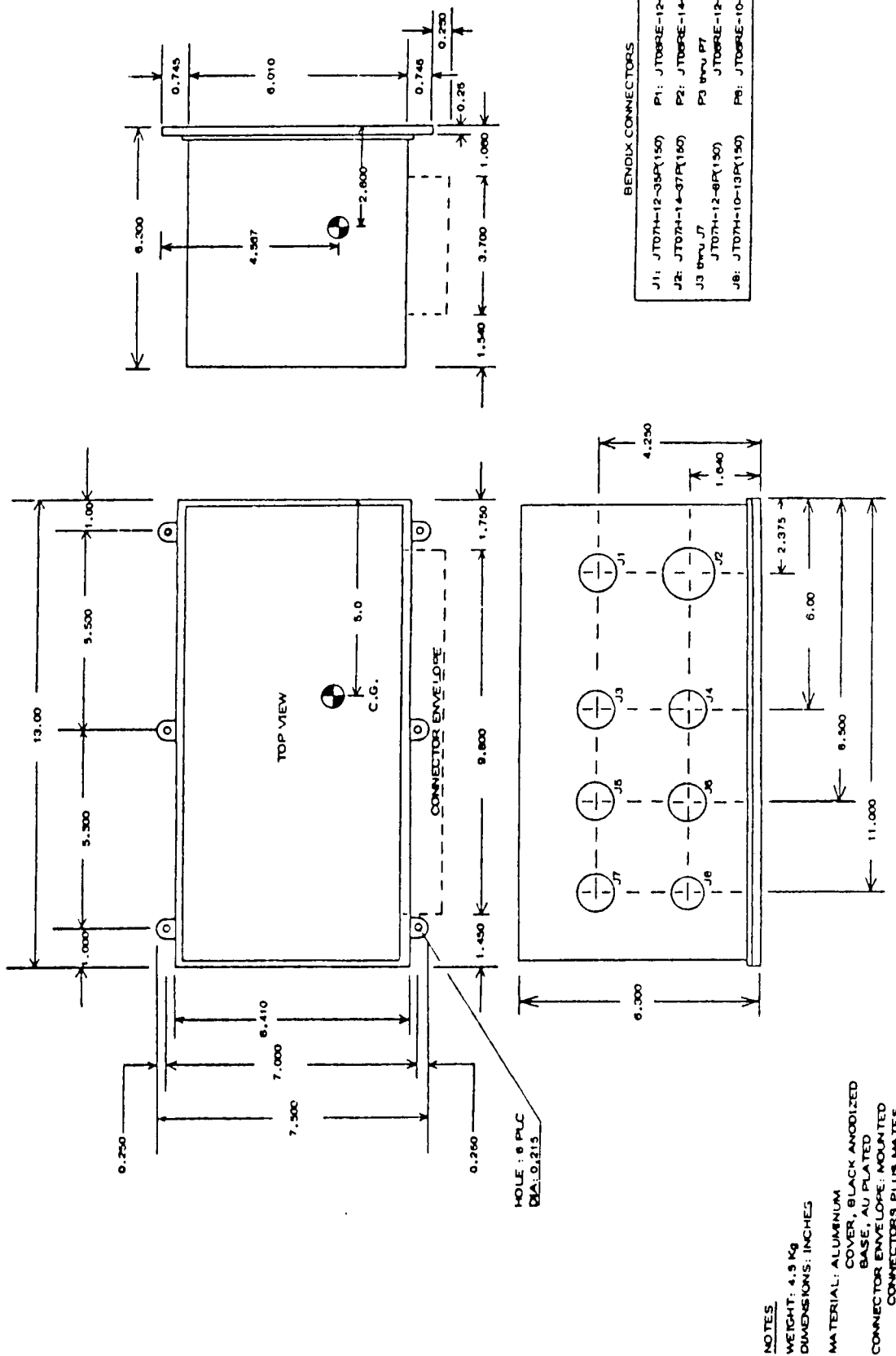


Fig. 2 TQCM Controller Mechanical Specifications

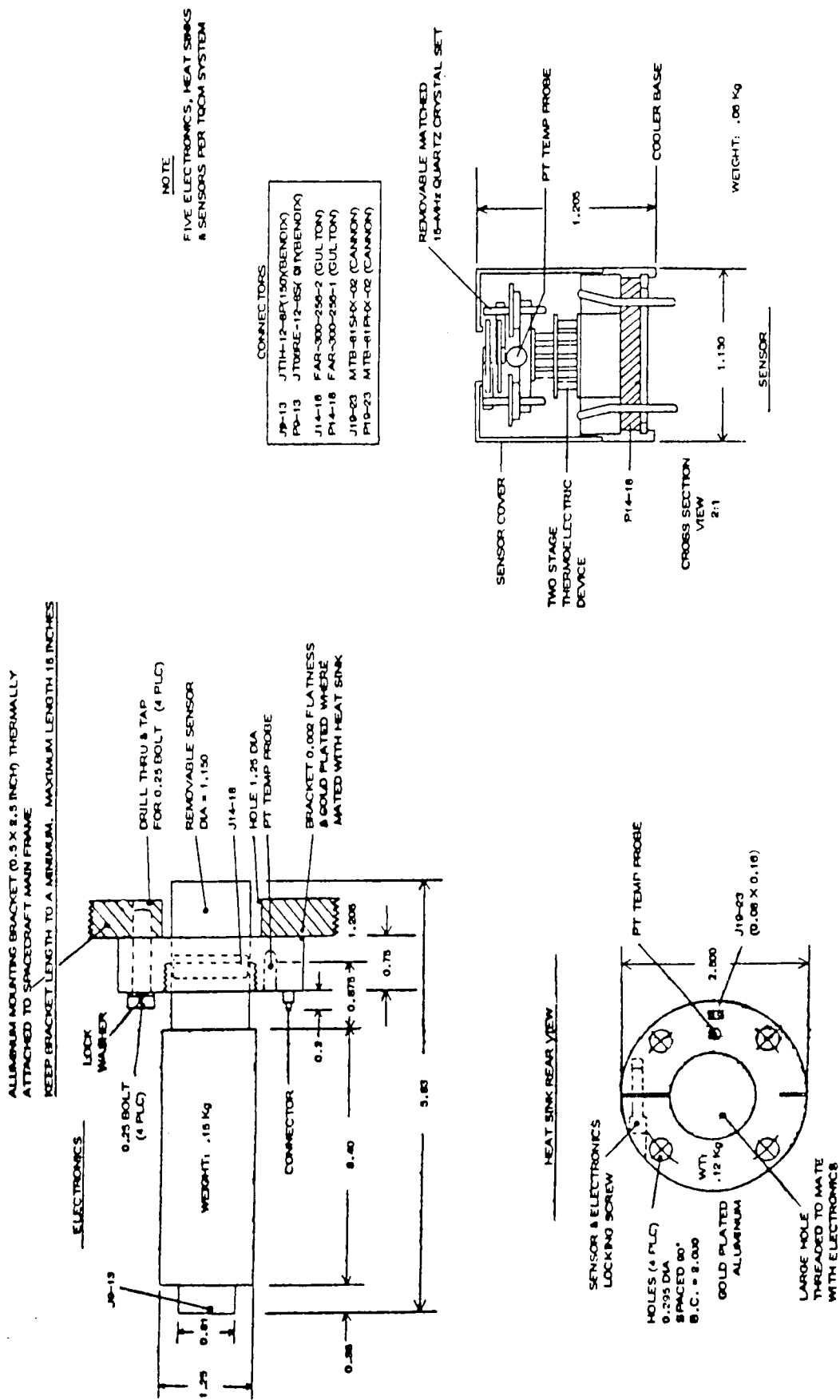


Fig. 3 TQCM Electronics, Heat Sink and Sensor Specifications

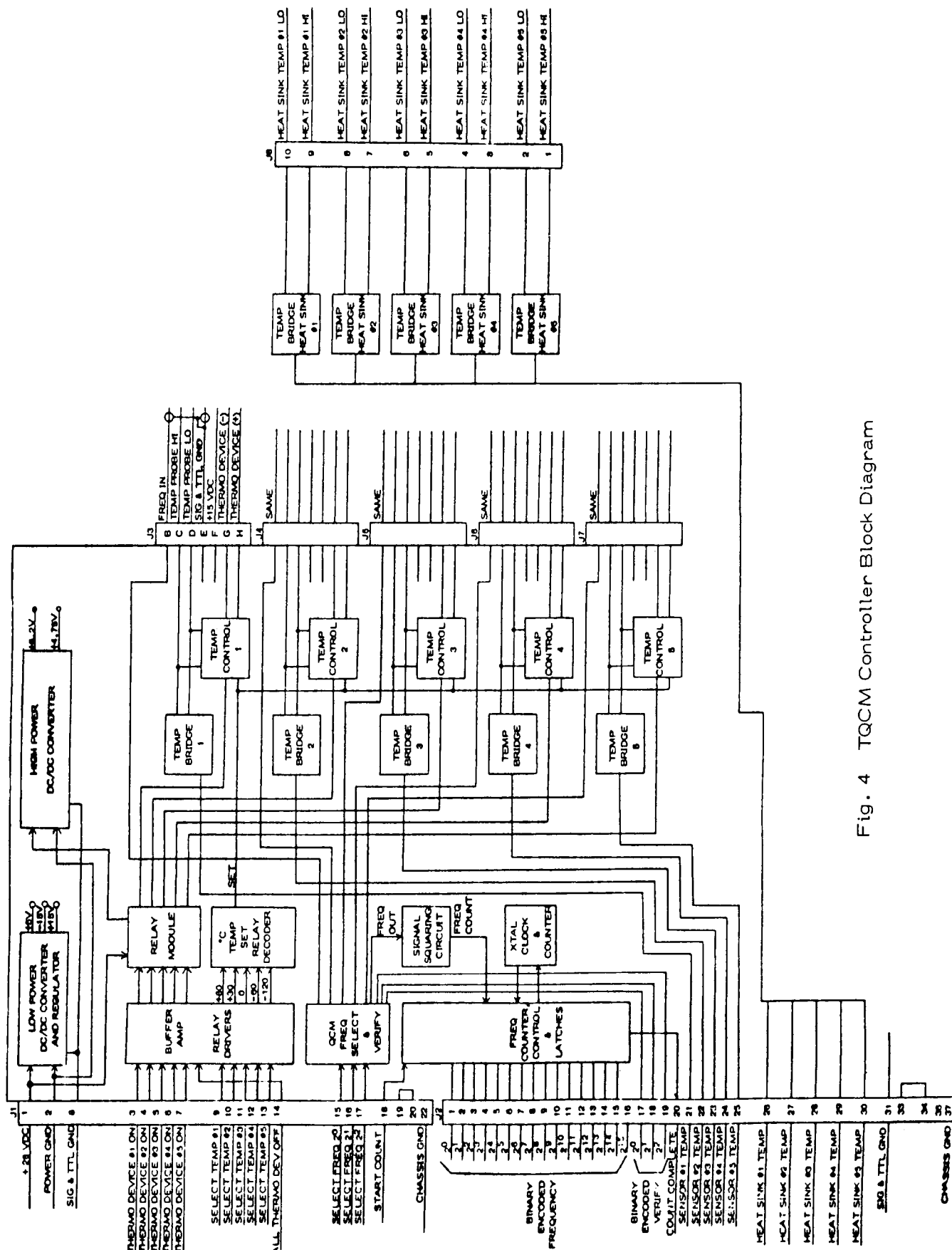


Fig. 4 TQCM Controller Block Diagram

Table 1 TQCM System Operating Powers

<u>Mode</u>	<u>Description</u>	<u>Pwr In</u>	<u>Inst Pwr</u>	<u>Eff %</u>
1. ON	All QCM's and Controller ON	1.85	1.11	60
2. Heat 1	All QCM's and Controller ON 1 Sensor heating maximum rate	5.44	2.72	50
3. Heat 5	All QCM's and Controller ON 5 Sensor heating maximum rate	13.35	8.68	65
4. Cool 1	All QCM's and Controller ON 1 Sensor cooling maximum rate	17.82	11.94	67
5. Cool 5	All QCM's and Controller ON 5 Sensor cooling maximum rate	71.12	54.76	77
6. Cool 5	All QCM's and Controller ON 5 Sensors stabilized -60°C	51.08	36.78	72
7. Cool 5	All QCM's and Controller ON 5 Sensors stabilized -30°C	28.93	20.25	70
8. Cool 5	All QCM's and Controller ON 5 Sensors stabilized 0°C	15.03	9.92	66
9. Heat 5	All QCM's and Controller ON 5 Sensors stabilized +30°C	7.15	3.93	55
10. Heat 5	All QCM's and Controller ON 5 Sensors stabilized +80°C	12.19	7.68	63

ting in a cooling mode at -30°C if there is a failure in the command data link. The -30°C mode was picked because of the relatively low power required to maintain cooling.

The TQCM response to temperature command is very rapid as is shown in Fig. 5. To cool a sensor from $+80^{\circ}\text{C}$ to -60°C requires 7 minutes and 3 minutes to return to $+80^{\circ}\text{C}$.

3.3 TQCM Laboratory Measurements

The TQCM was tested in a laboratory vacuum chamber at 7×10^{-7} Torr by measuring the contamination of a surface by roughing pump oil. The adsorption and desorption of oil by an aluminum surface is shown in Fig. 6. Below -30°C absorption predominated and contamination built up. Above -30°C desorption predominated. The break in the curve at about -10°C resulted from a phase change in the contamination from solid to liquid.

The number of monolayers of contamination on the aluminum surface with temperature is shown in Fig. 7. The surface was first cleaned at $+80^{\circ}\text{C}$ and rapidly dropped to -20°C . The contamination loading was then allowed to reach equilibrium at various higher temperatures. From the figure, it can be seen that there was always oil contamination present below $+30^{\circ}\text{C}$. It would be impossible to have made these measurements without a temperature-controlled QCM because there is no net mass change at equilibrium.

Data similar to Fig. 6 and 7 should be seen on Shuttle but of a much more complex nature because of the large variety of contaminants present. Solar radiation of the contaminated surface and upper atmospheric interactions will further complicate the analysis.

4.0 CQCM SYSTEM

4.1 CQCM Design

The CQCM is radiatively cooled and operates at cryogenic temperatures⁵. It is designed to monitor highly volatile contaminants. The CQCM, consisting of a Controller and Sensor Head, is shown in Fig. 8. Measurements are made with two removable 15-MHz mass sensors mounted in the radiant cooler. One sensor operates below the freezing point of water which is approximately -133°C (140°K) in vacuum. It monitors contamination including that of water vapor. The second sensor is heated above the freezing point of water and monitors the contamination background. It provides a

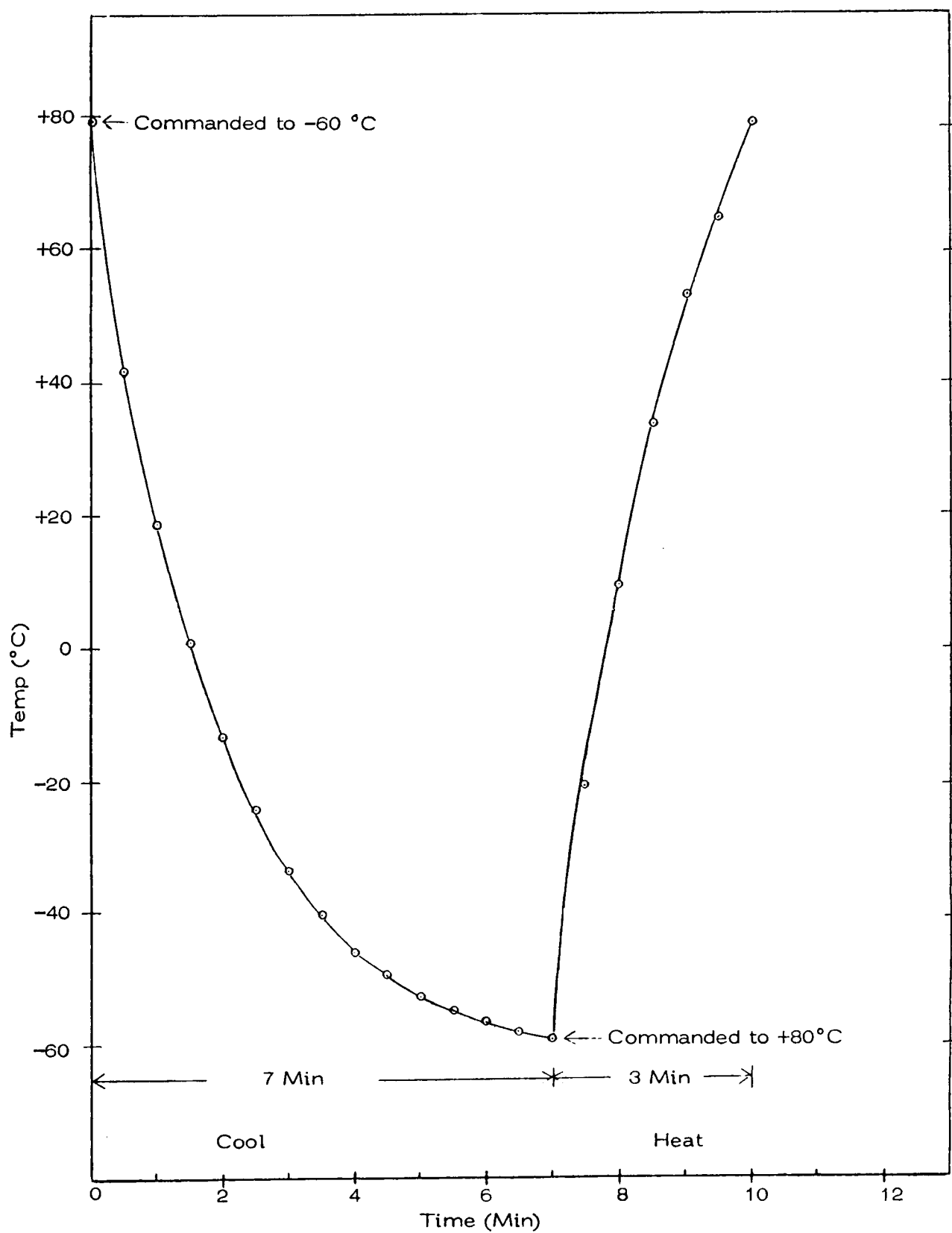


Fig. 5 Times Required for TQCM to Reach -60 °C and +80 °C for Shuttle Operation (+20 °C Heat Sink)

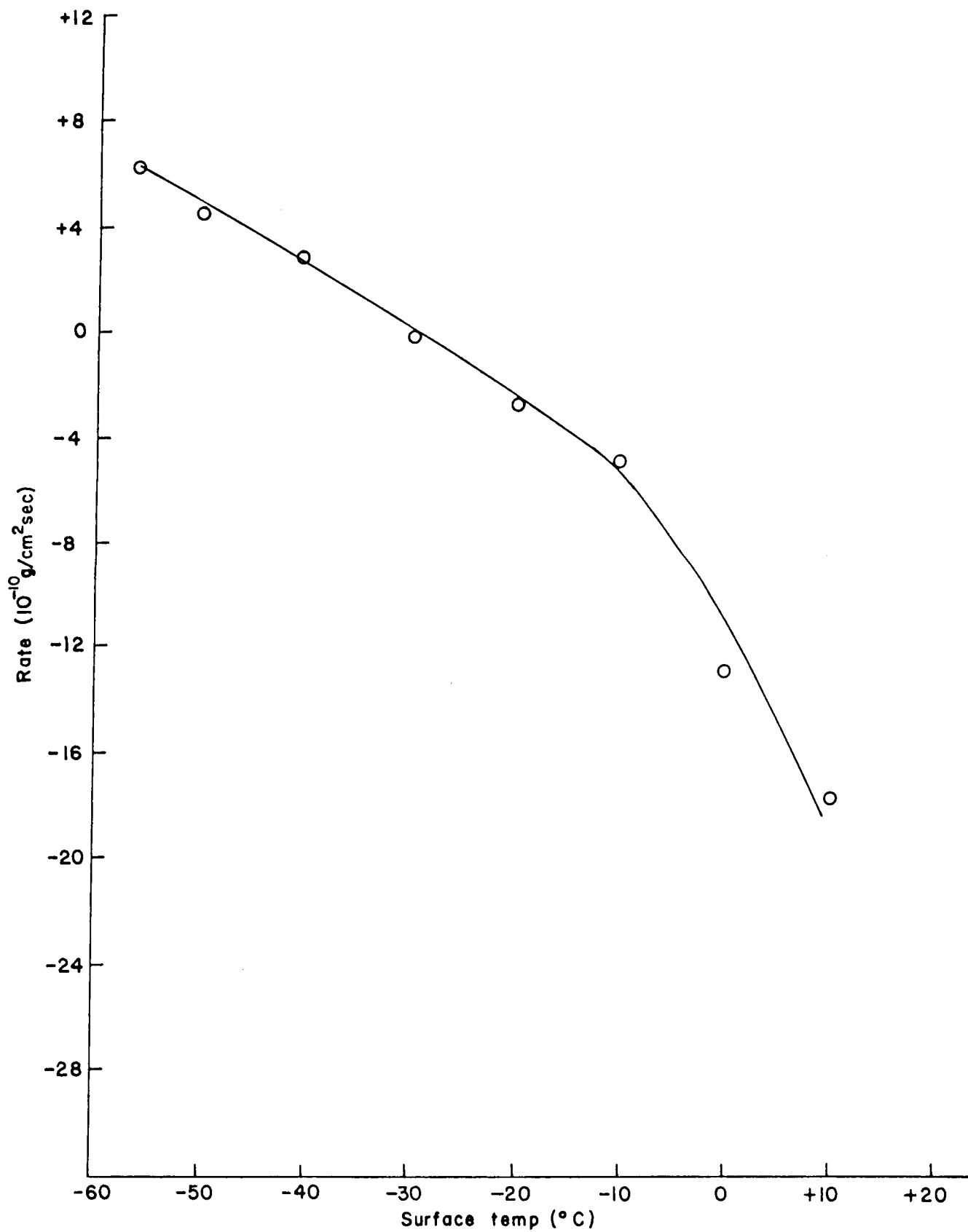


Fig. 6 Contamination adsorption and desorption rates with temperature for roughing pump oil on aluminum in vacuum

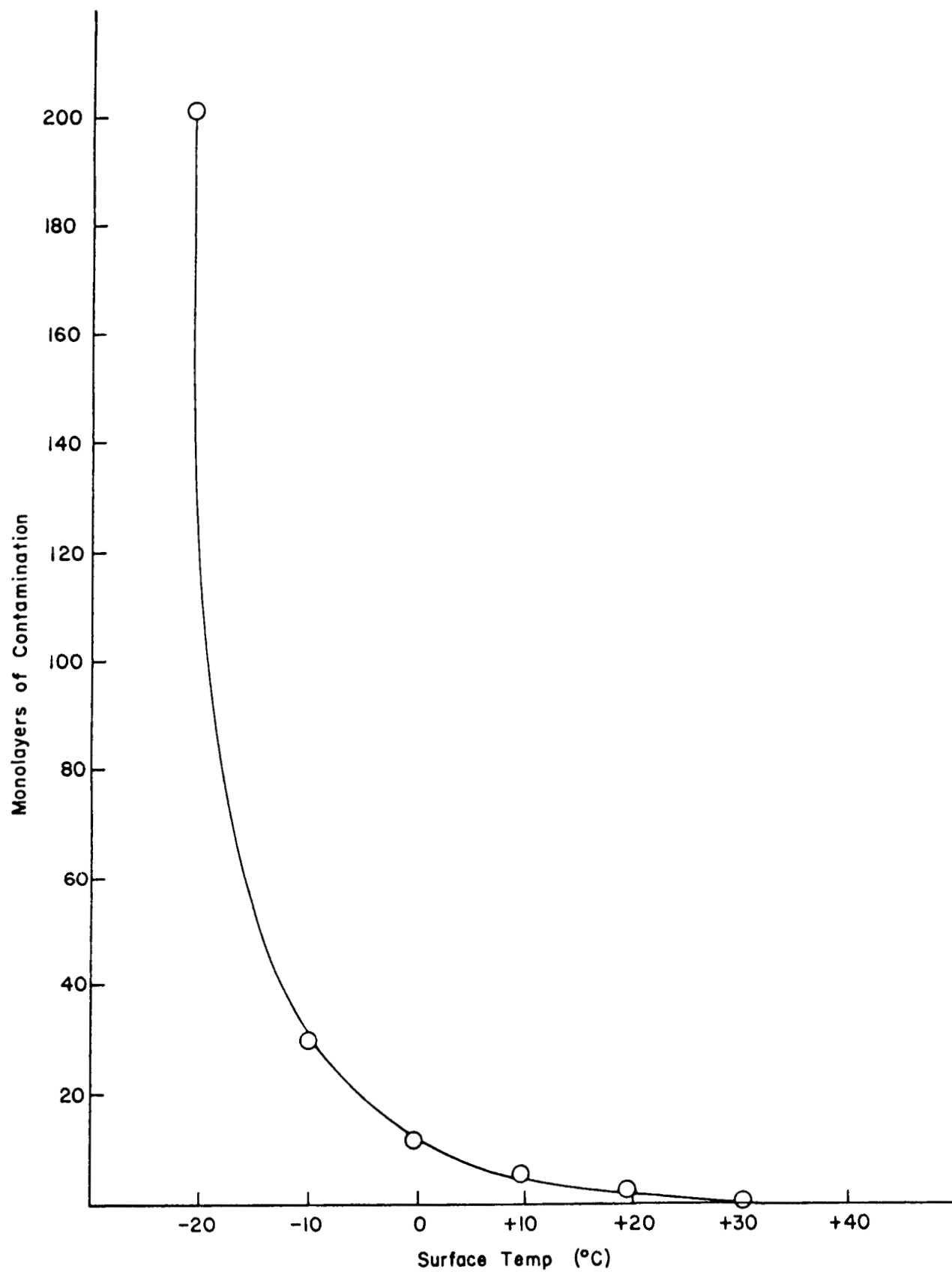


Fig. 7 Number of contamination monolayers of roughing pump oil on aluminum at equilibrium in vacuum

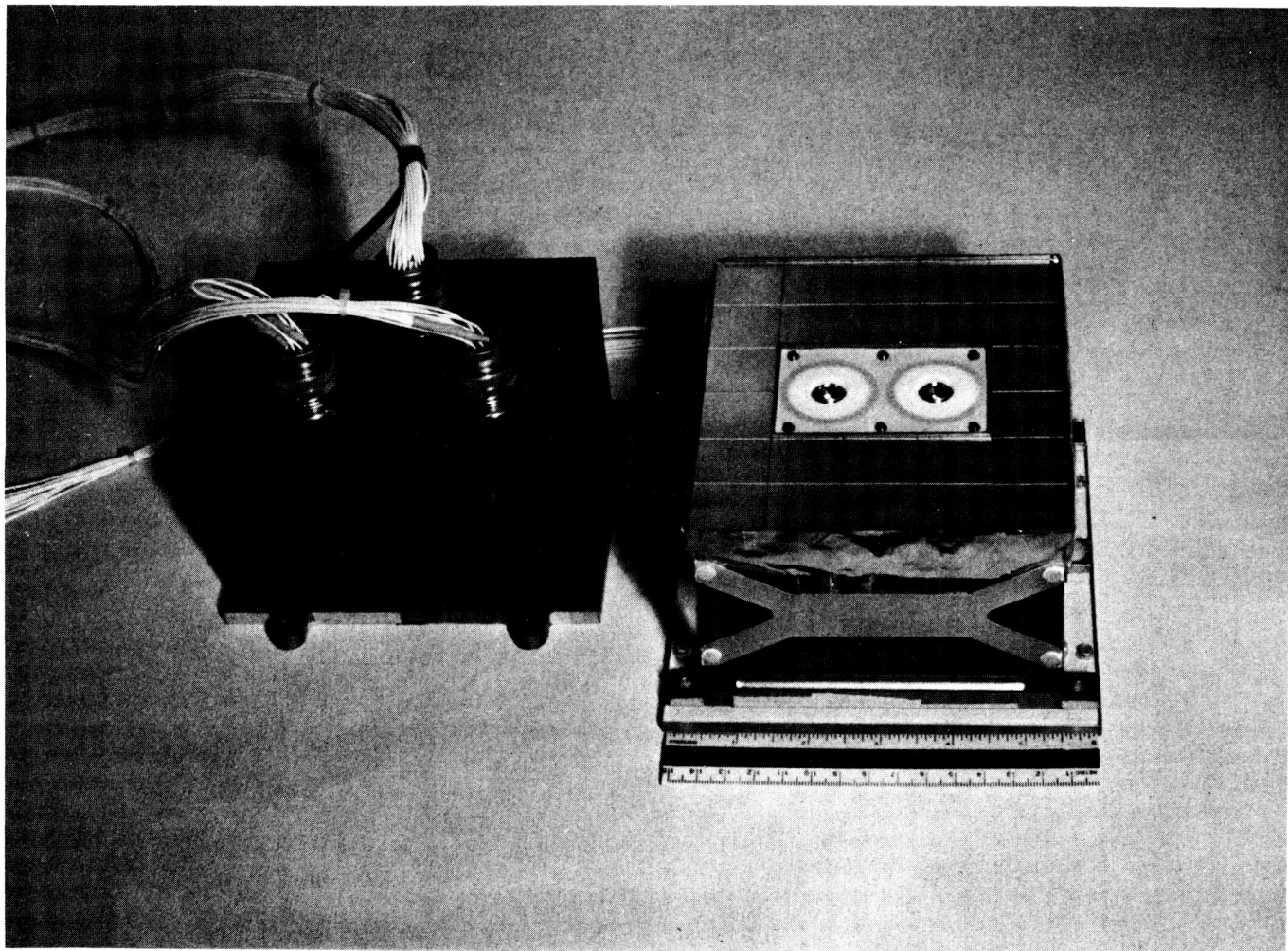


Fig. 8 CQCM System

reference from which the density of the water vapor cloud enveloping Shuttle can be determined. The mass measurement range of each sensor is the same as the TQCM. The temperature of each sensor is monitored between -153 and $+37^{\circ}\text{C}$ (120 and 310°K). The design incorporates a low-power electronics⁴ and heaters for ice removal.

The CQCM Controller ($13.21 \times 14.99 \times 8.41$ cm) mechanical specifications are shown in Fig. 9.

The CQCM Sensor Head ($15.24 \times 16.15 \times 8.41$ cm) design is shown in Fig. 10. The Sensor Head contains the radiant cooler, two removable mass sensors, oscillators and output amplifiers.

The radiant cooler is a plane surface constructed from one piece of aluminum. The sensors are mounted in a well, machined in the radiator. Second-surface quartz/silver mirrors are attached to the radiator by an RTV. The mirrors have a solar absorptance of approximately 0.06 and a thermal emittance of approximately 0.80.

The radiator is attached to the base plate by four high-strength fiberglass-epoxy laminate standoffs. The two electronic units are connected to the mass sensors by stable ohm leads to provide thermal isolation. Radiative heat exchange from spacecraft to the radiator is minimized using a stack of 20 multilayer superinsulation of gold plated Kapton.

The CQCM Controller Block Diagram is shown in Fig. 11. The Controller provides DC/DC isolated power to operate the CQCM. Frequency of each sensor is readout by command thru a $65,535 \text{ Hz} \pm 1 \text{ Hz}$ capacity counter. Temperature bridges 1 and 2 readout the water vapor and reference sensor temperatures. Inputs are provided for turning ON the radiator and sensor heaters for ice removal.

The remotely located Sensor Head is connected to the Controller at J2 and 3.

4.2 CQCM Operation

The CQCM commanded modes of operation are shown in Table 2. It operates on $+28 \pm 4 \text{ Vdc}$. Minimum power is 1.33 W with only the QCM's ON. Maximum power of 3.08 W occurs with both QCM and all heaters ON.

4.3 CQCM Thermal Modeling

The operating temperatures were obtained by computer thermal modeling⁵.

The calculated cool-down time of the CQCM with only the Refer-

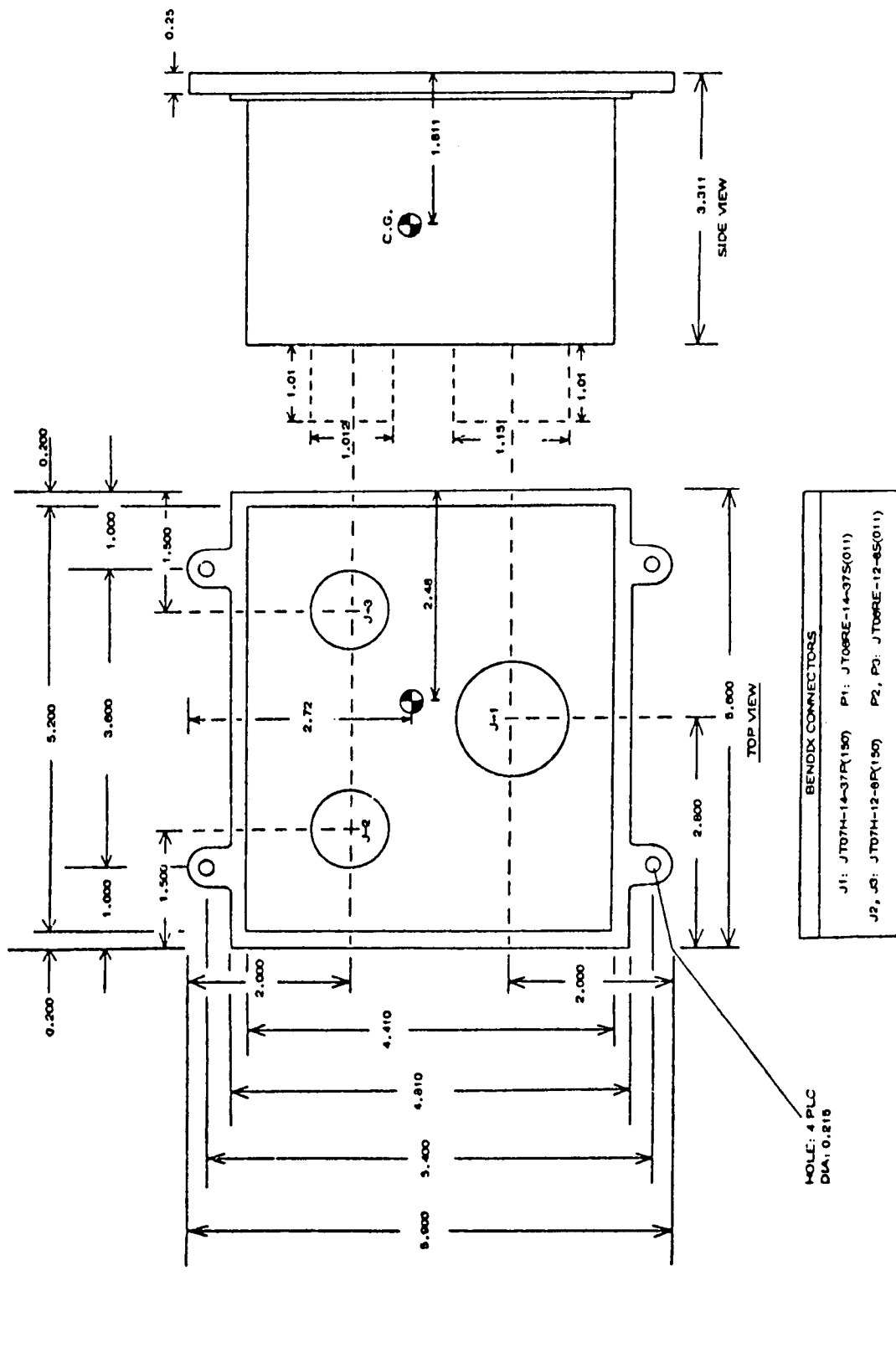


Fig. 9 CQCM Controller Mechanical Specifications

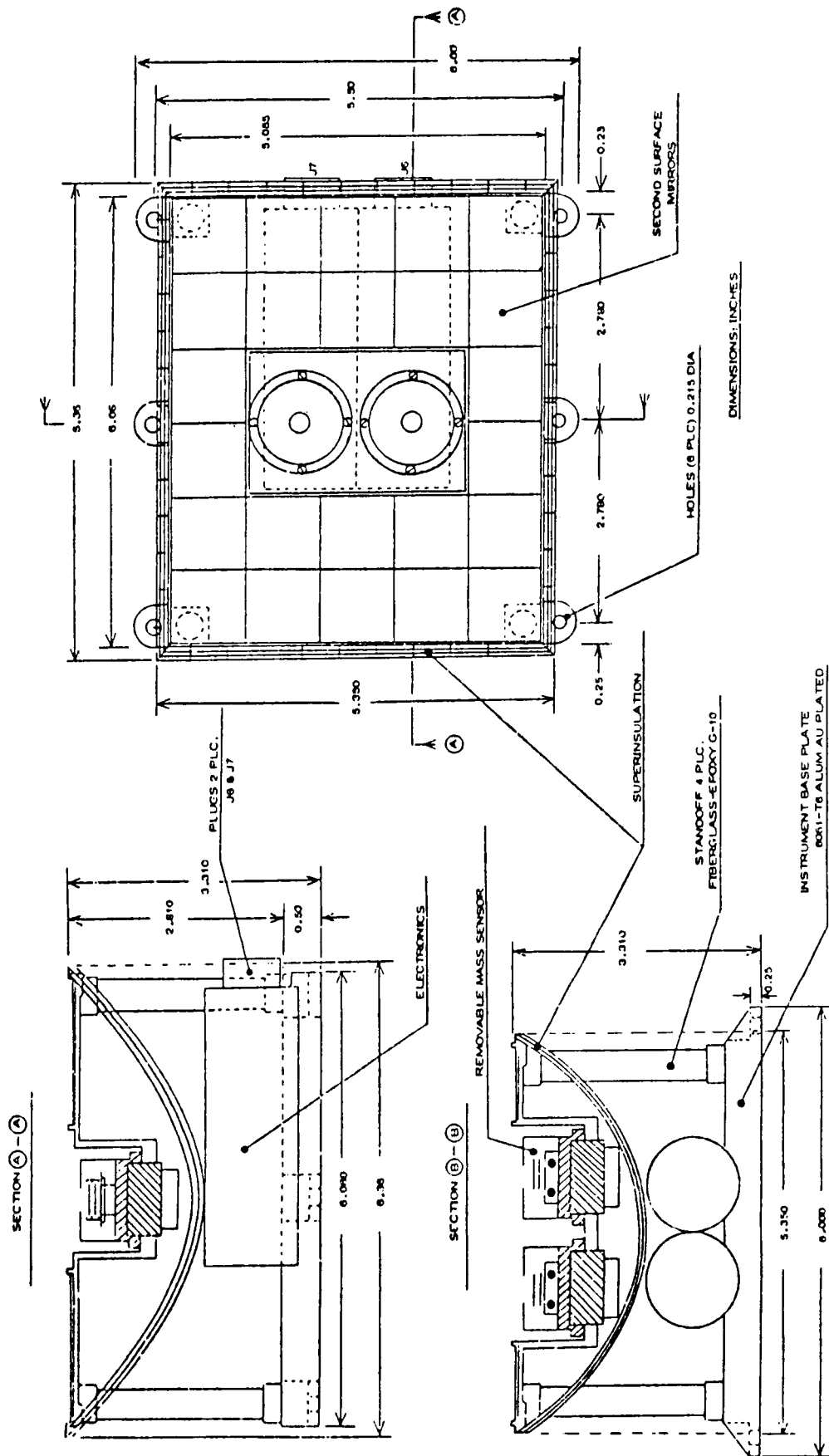


Fig. 10 QCM Sensor Head Specifications

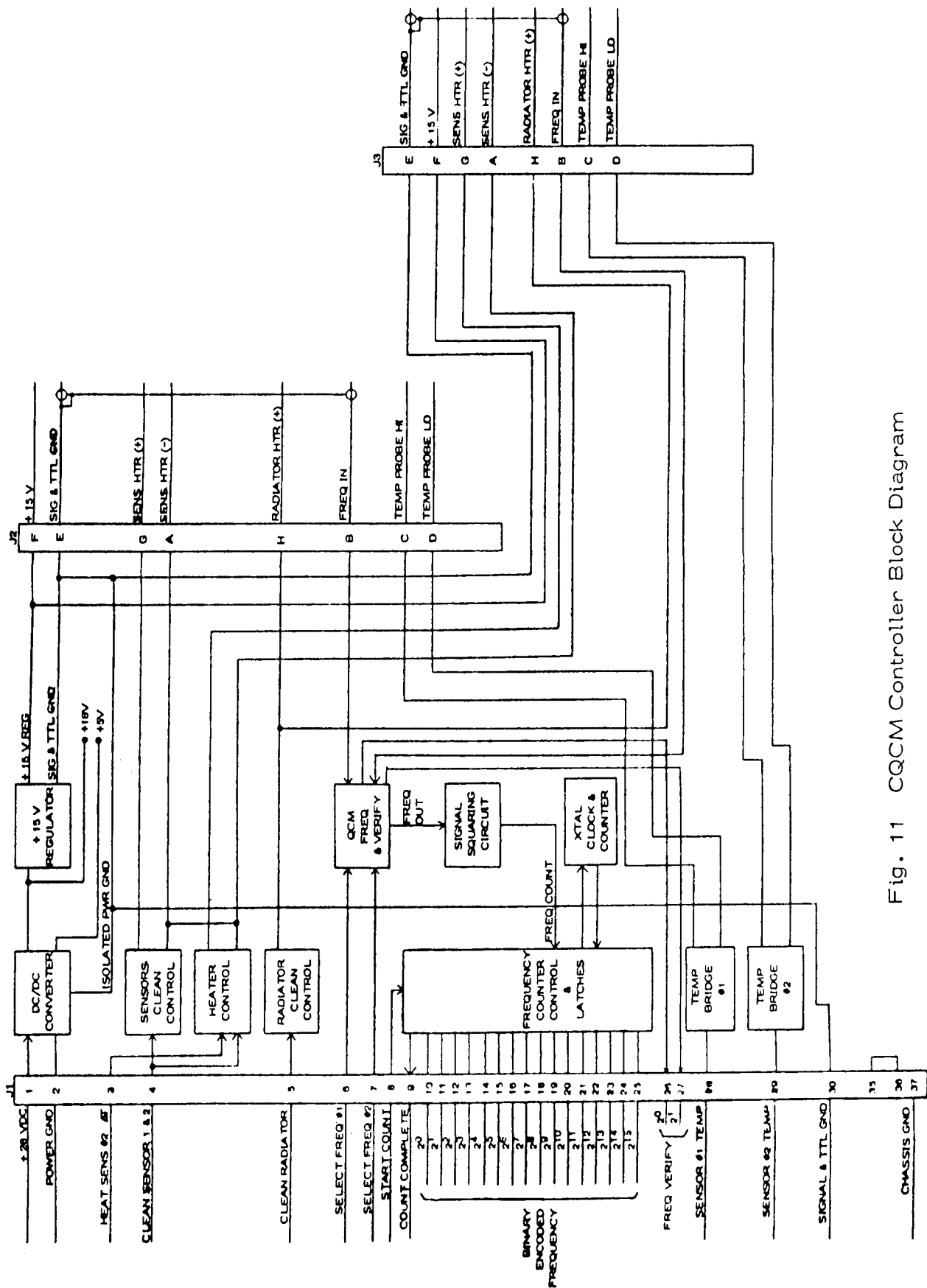


Fig. 11 CQCM Controller Block Diagram

Table 2 CQCM System Operating Powers

<u>Mode</u>	<u>Description</u>	<u>Pwr In</u>	<u>Inst Pwr</u>	<u>Eff %</u>
1. Static (minimum)	QCM's and Controller ON	1.33	.60	45
2. Normal Operation	QCM's and Controller ON Ref Sensor heater ON	1.84	.92	50
3. Clean 1 & 2	QCM's and Controller ON Water Vapor Sensor Heater ON	2.28	1.30	57
4. Clean radiator	QCM's and Controller ON Radiator Heater ON	2.04	1.14	56
5. Clean all	QCM's and Controller ON All Heaters ON	3.08	1.85	60

ence Sensor heated is shown in Fig. 12.

The calculated temperature rise of the CQCM with power to the water vapor monitor radiant cooler for ice removal is shown in Fig. 13.

5.0 SYSTEMS TEST MONITOR

A monitor has been built to functionally test the operation of the CQCM and TQCM System. The monitor is shown in Fig. 14 with CQCM and TQCM Systems in place for functional tests.

The Monitor provides a simple to operate, direct method for check-out and functional test of the TQCM or CQCM System. At the launch site, it will provide a rapid check of proper instrument functioning or a quick test of sensor operation after contaminated sensors are replaced without having to use the spacecraft telemetry system.

The Monitor has displays or switches for testing every active pin function on the TQCM or CQCM System. The Monitor specifications are

- 1) Power: 115 V, 2A (60 Hz)
- 2) Provides all required DC power for operating a TQCM or CQCM System.
- 3) The frequency output of any TQCM or CQCM mass sensor can be readout by selecting the proper binary coded panel switches. When switches are operated, lamps verify the commands.
- 4) The digital frequency meter displays output of any desired TQCM or CQCM mass sensor. A voltage analog of frequency is provided as an output for recording.
- 5) The digital temperature meter displays temperature of any desired TQCM Sensor or Heat Sink or CQCM Sensor temperature. A voltage analog of temperature is provided as an output for recording.
- 6) A ten position switch is used to set up a particular TQCM Sensor Temp (1 thru 5) or Heat Sink Temp (6 thru 10).
- 7) Total current meter allows various TQCM or CQCM operating modes to be monitored for possible deviation from design power values. An analog output is provided for recording current.
- 8) LED lamps indicate whether the TQCM or CQCM is being tested.
- 9) Command switches provide temperature inputs to the TQCM or CQCM to simulate Shuttle flight operations.

6.0 ACKNOWLEDGMENTS

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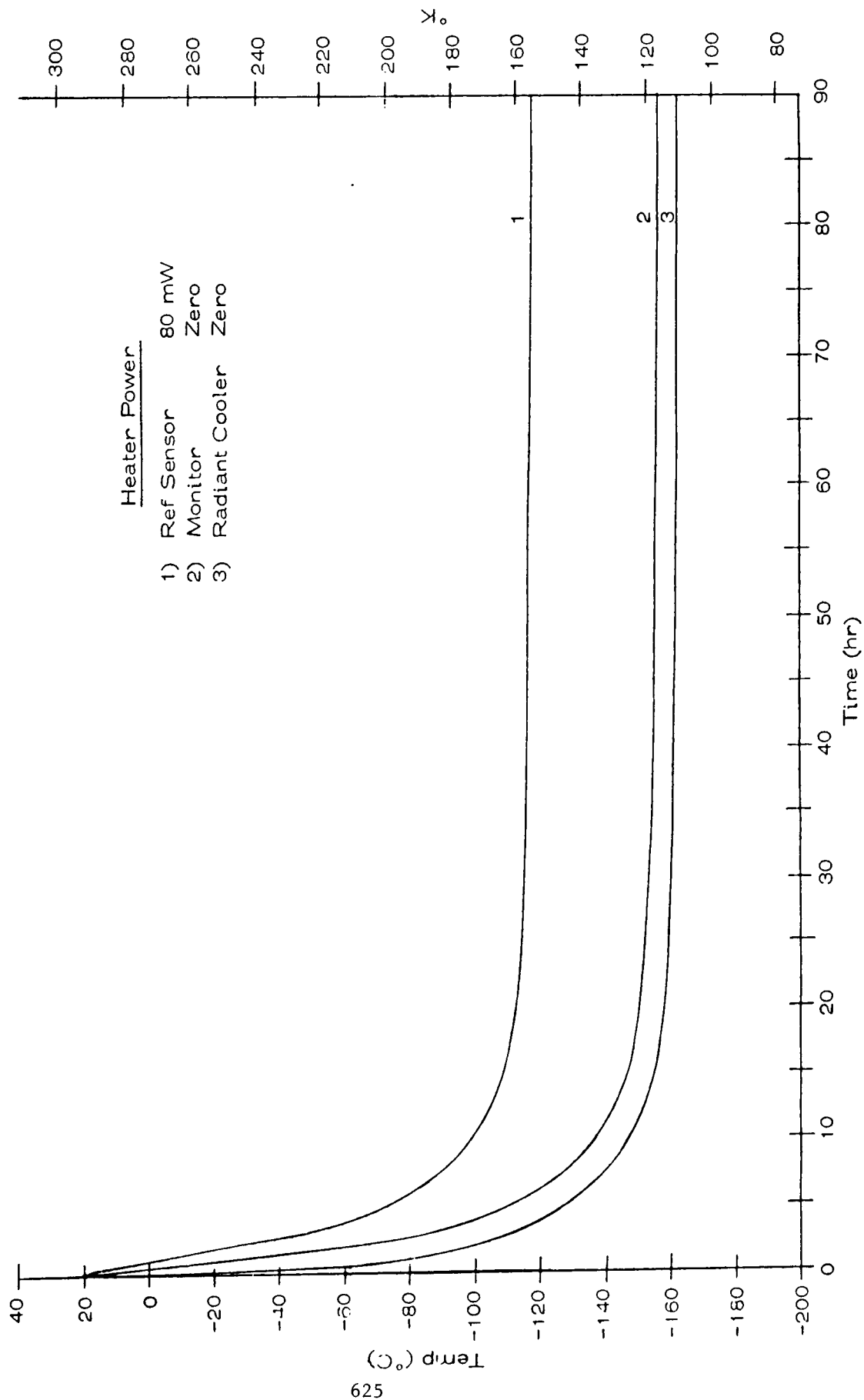


Fig. 12 Calculated CQCM Cool-Down with Power-On in Space

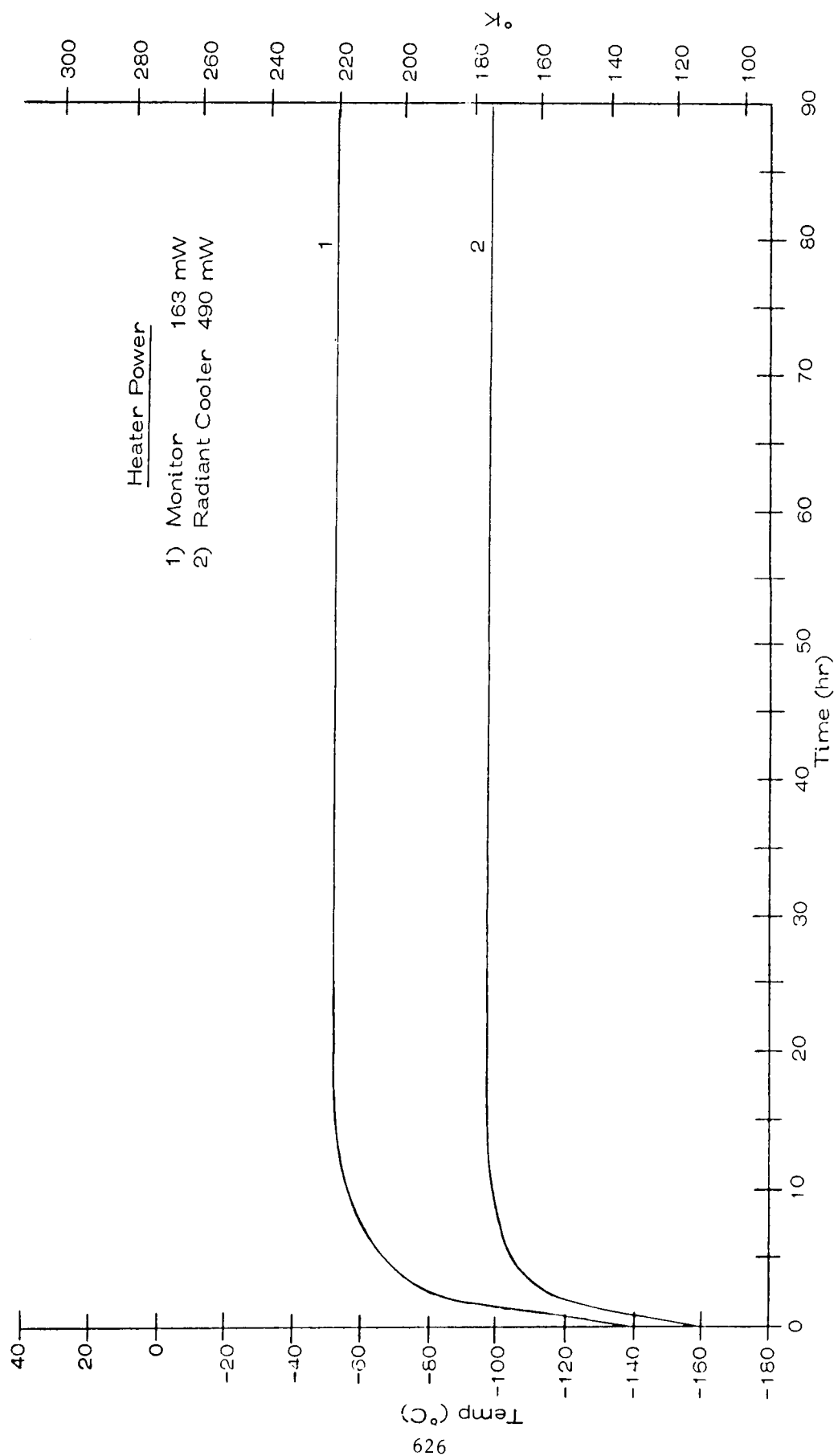


Fig. 13 Calculated CQCM Temperature in Space when Heaters are Operated for Ice Removal

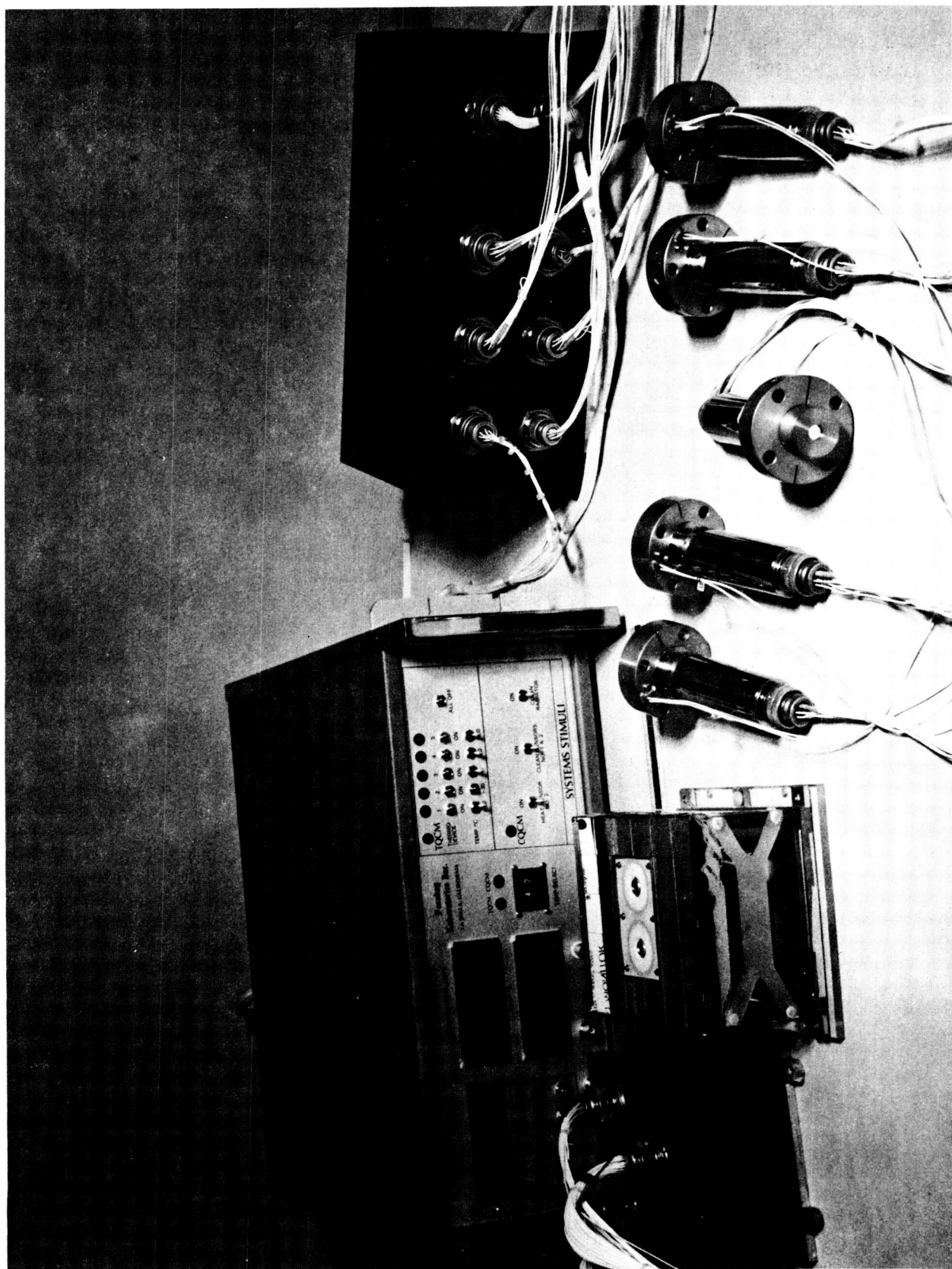


Fig. 14 Functional Tests of CQCM and TQCM Systems

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7.0 REFERENCES

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